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NATIONAL WATER-QUALITY ASSESSMENT PROGRAM

FIPRONIL AND DEGRADATION PRODUCTS IN THE RICE-PRODUCING AREAS OF THE MERMENTAU RIVER BASIN, LOUISIANA, FEBRUARY-SEPTEMBER 2000

Significant Findings

- Maximum fipronil concentrations in water ranged from 0.829 to 5.29 µg/L (micrograms per liter) during March through April, corresponding to the release of ricefield tailwater.
- Fipronil degradation products in water were detected from February through June 2000. Aerobic degradation products desulfinylfipronil and fipronil sulfone were detected at maximum concentrations in March and April, similarly to the parent compound. Maximum concentrations of the anaerobic degradation product fipronil sulfide were detected in June.
- Concentrations of fipronil and degradation products in suspended sediment ranged from 1 to 10 percent of the concentrations in water.
- The parent compound fipronil is not accumulating in bed sediment.
- The anaerobic reduction degradation product fipronil sulfide was the predominant degradation product detected in bed sediment, and concentrations ranged from 0.636 to 24.8 µg/kg (micrograms per kilogram). The degradation products desulfinylfipronil and fipronil sulfone also were frequently detected in bed sediment.

INTRODUCTION

The Mermentau River Basin is a unique agricultural area located in southwestern Louisiana. Historically, the area was a tallgrass prairie similar in many ways to the tallgrass prairie in the midwestern United States (Allain and others, 2000). The area also is called coastal prairie or wet prairie (Brown, 1972). The distinctive prairie characteristics of the basin are a result of an impermeable clay layer underlying the shallow soils. Five major tributaries divide the basin into a series of broad, flat areas ideal for agriculture. The areas are separated by bottomland hardwood riparian corridors that vary in width from only a hundred feet to several miles. Thus, the basin is characterized by a dendritic river system, low gradient, and a heavy clay soil.

Most of the Mermentau River Basin has been altered for growing rice, sugarcane, forage, grain crops, crawfish production, and cattle grazing. Rice, however, is the most important agricultural crop produced in the basin. Crawfish farming also occurs in ricefields. The insecticide fipronil was licensed in 1996 (U.S. Environmental Protection Agency, 1996a), and the insecticide was widely used on rice seed to control the rice water weevil beginning in 1999. In the summer of 1999, public concern in Louisiana arose over the use of fipronil-coated rice seed and the apparent negative effects on commercially-produced crawfish populations in the Mermentau River Basin. Traditional rice-cultivation practices include releases of ricefield water in the spring months. The effects of ricefield drainage on receiving waters and biota are not well understood.

This report presents a study of the analyses of fipronil and three of its degradation products in water, suspended sediment, and bed sediment. Samples were collected from 17 sites in the rice-producing areas of the Mermentau River Basin from February to September 2000 collected as part of the U.S. Geological Survey National Water-Quality Assessment (NAWQA) Program. This insecticide and its degradation products--desulfinylfipronil, fipronil sulfide, and fipronil sulfone--were selected for discussion because little information currently exists on the occurrence and partitioning of this new insecticide in the environment.

The Mermentau River Basin has a drainage area of about 3,800 mi^2 (square miles) (Sloss, 1971). Land-surface elevations are less than 100 feet in most of the basin and range from 20 to 30 feet above the National Geodectic Vertical Datum of 1929 along the Mermentau River main stem (Garrison, 1997). Vegetation in the upper basin consists of mixed pine-hardwoods, but as the land flattens vegetation rapidly changes. The Mermentau River Basin in the mid-to-lower reaches is bordered by a cypress-water tupelo riparian zone that is typically flooded for a large part of the year. The banks generally are steep in the tributaries with cypress-water tupelo extending into the stream. Along the lower reaches, banks often are not discernible as the water extends into a bottomland hardwood flood plain. The flood plain may abruptly end as it meets a ricefield levee system or roadway. Water velocities are typically less than 1 ft/s (foot per second). Isolated flow reversals can occur in the tributaries and canals draining into the river during periods of heavy pumping for rice irrigation. The streambed substrate in the tributaries and main channel is silt and clay. The substrate in the loops of the main stem and smaller tributaries can also be a soft flocculent muck rich in organic matter.

Total rice acreage in the seven parishes (Acadia, Allen, Evangeline, Jefferson Davis, Lafayette, St. Landry, and Vermilion) of the Mermentau River Basin was almost 400,000 acres in 1998 and about 340,000 acres in 2000 (Louisiana Cooperative Extension Service, 1998, 2000). The primary cause of the decrease in rice acreage was a drought in south Louisiana which began in the spring of 1999 and lasted well into the summer and fall of 2000. Rice cultivation in this basin utilizes both ground and surface water (fig. 1) (B.P. Sargent, U.S. Geological Survey, written commun., 2002). The decreased availability of fresh surface water during the drought in the southern part of the basin, especially Vermilion Parish, resulted in less rice planted in 2000 than in 1998.

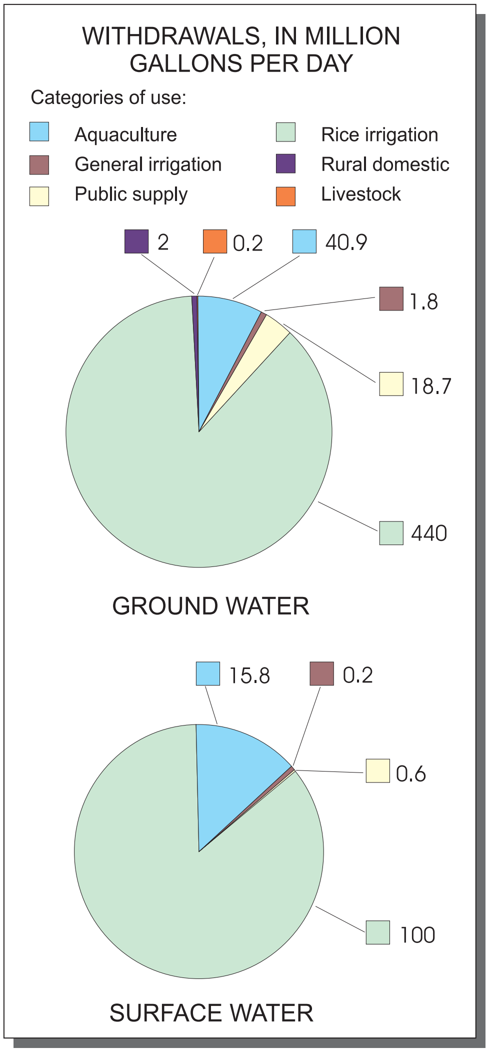

Figure 1. Ground- and surface-water withdrawals in the Mermentau River Basin, Louisiana, 2000. (Data from: B.P. Sargent, U.S. Geological Survey, written commun., 2002.)

Aerial application of seed onto flooded fields is the predominant method of rice seeding in southwestern Louisiana. After aerial rice seeding, the water is retained for about 1 to 2 days and released. The field is allowed to drain only long enough for the young seedling to become anchored, about 3 to 5 days. The field is then re-flooded until the rice nears maturity. Releases of ricefield water (called tailwater) into drainage ditches and streams result in high turbidities during March through May.

Preparation for aerial-rice seeding in the Mermentau River Basin.

The flooding of fields also functions to suppress red rice, a wild variety with no commercial value (Linscombe and others, 1999), and to make ricefields easily adaptable for crawfish farming. Most crawfish farming occurs in ricefields, either double-cropped with rice or utilizing ricefields that are not in rice production that year. There are more than 80,000 acres of farm-raised crawfish in southern Louisiana, including about 40,000 acres in the Mermentau River Basin in 2000.

Study Design

Prior to the public concern about fipronil, an assessment of agricultural practices on stream quality and aquatic invertebrates in the Mermentau River Basin was designed as part of the NAWQA Program. The purpose of the assessment is to integrate surface- and ground-water-quality data with information about aquatic invertebrate community structure to gain a more complete understanding of the factors, both natural and human-induced, that affect water quality in the Mermentau River Basin. The community structure of aquatic invertebrates in this area is of interest because, in addition to their value as indicators of the overall health of the system, the ricefields of southwestern Louisiana are an internationally important stopover site for invertebrate-eating shorebirds during their spring migration. About 3,200 mi^2 of the Mermentau Basin are assessed by NAWQA. Three major components of the assessment have been completed, as follows:

- 2000: Surface-water quality--Occurrence and distribution of major inorganic ions, nutrients, and pesticides in water; also fipronil and degradation products in water, suspended sediment, and bed sediment.
- 2000: Ground-water quality--Occurrence and distribution of major inorganic ions, nutrients, trace elements, and pesticides including fipronil and degradates in shallow ground water.
- 2001: Ecology and aquatic invertebrate community structure--Integration of chemical data with ecological data including aquatic invertebrates.

Site Selection

The sampling sites for the 2000 surface-water-quality component of NAWQA (fig. 2) were selected to represent the water quality at 2 or 3 locations from each major tributary: Bayou des Cannes, Bayou Nezpique, Bayou Lacassine, Bayou Plaquemine Brule, and Bayou Queue de Tortue (table 1). One site was located near the headwaters of each major tributary and 1 to 2 sites were located mid-reach or downstream. Two sites were located on the Mermentau River main stem: Mermentau at Mermentau and Mermentau at Lake Arthur. Mermentau at Mermentau is a long-term USGS water-quality site. Downstream from Mermentau at Lake Arthur, tidal effects and the Gulf Intracoastal Waterway greatly complicate data analysis and were excluded from this study of fipronil and three of its degradation products. The sites were sampled monthly February to June 2000. Bed sediment samples were collected from August 28 to September 6, 2000.

Sample Collection and Laboratory Analysis

USGS personnel conducted extensive discussions with the following organizations before sampling for fipronil and its degradation products: Louisiana Department of Agriculture and Forestry, Louisiana State University Agriculture Center, including their Aquaculture Research Station and Rice Research Station, and Bayer CropScience, the manufacturers of fipronil.

Water column samples were collected according to methods described in Fishman and Friedman (1989) and Shelton (1994). In those streams where water velocities were less than 1.5 ft/s, a weighted bottle sampler was used to collect a depth-integrated sample. Samples were processed on site in a USGS mobile laboratory. For analysis of fipronil and degradation products on suspended sediment, the volume of water passing through a baked glass-fiber filter was measured, and the glass-fiber filter was retained and frozen until analysis. Fipronil and degradation products were analyzed at the USGS National Water Quality Laboratory in Lakewood, Colorado. These compounds were not part of any standard USGS analyses prior to this study. Personnel at the USGS laboratory developed a custom method for these analyses in filtered water. This method was a modification of an approved analytical method (Zaugg and others, 1995). Bed sediment samples were collected using a Petite Ponar grab sampler. This sampler has a 6-by-6 inch collection area and penetrates about 4 inches into the bed sediments. Five bed-sediment samples were collected at each site, composited, and a subsample was placed in a baked glass bottle and shipped at 4 degrees Celsius to the laboratory. The sediment and suspended-sediment methods were custom modifications of a method for determinations of compounds in bed sediment (Furlong and others, 1996).

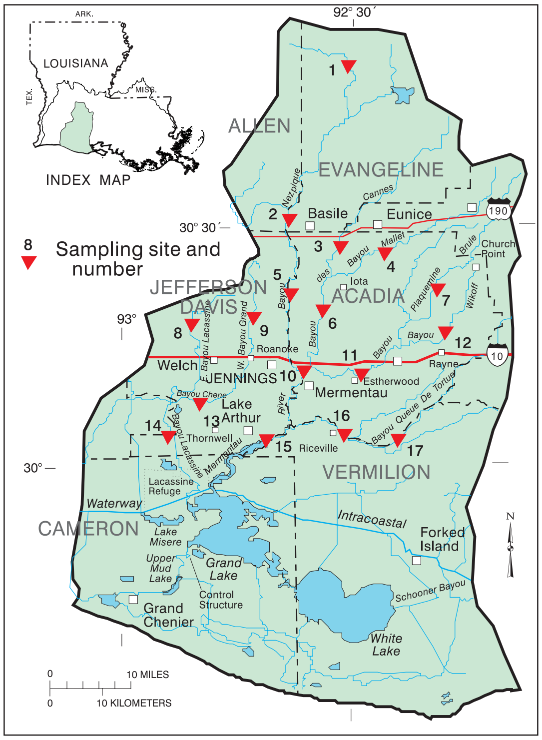

Figure 2. Study area in the Mermentau River Basin, Louisiana. (Modified from Garrison, 1997, p. 15.)

FIPRONIL AND DEGRADATION PRODUCTS

Fipronil, 5-amino-1-[2,6 dichloro-4-(triflurомethyl)phenyl]-4-[(1R,S)-(trifluromethyl)sulfinyl]-1H-pyrazole-3-carbonitrile, was discovered in 1987 and first registered as a pesticide in the United States in 1996. Fipronil is a phenylpyrazole insecticide which functions by targeting the gamma-aminobutyric acid (GABA) receptor system of insects (Kidd and James, 1991), interfering with chloride transport through the membranes of neurons. At sufficient doses, fipronil causes neural excitation, paralysis, and insect death (Bobe and others, 1998).

As organic compounds, pesticides have an affinity for the natural organic matter present in soils or water (Barbash and Resek, 1996). This affinity can be expressed by reporting a coefficient K indicating the compound's organic carbon partitioning coefficient (K_{oc}) or octanol/water partitioning coefficient (K_{ow}). The larger the K_{oc}, the greater the affinity of the compound to bind to soil particles, resulting in lower mobility through the soil. Based on the K_{oc} data (table 2), fipronil and desulfinylfipronil are expected to display low-to-moderate mobility, and fipronil sulfide and fipronil sulfone a low mobility (Connelly, 2001). The fipronil parent compound has a log K_{ow} of 4.01, which places it between highly insoluble pesticides such as DDT, which has a log K_{ow} of 6.2, and highly soluble pesticides such as atrazine, which has a log K_{ow} of 2.6 (U.S. Environmental Protection Agency, 1996b).

These chemical characteristics indicate that fipronil and its degradation products have differing mobilities and environmental fates. Therefore, to document the presence of these compounds in the environment it is necessary to analyze water, suspended sediment, and bed sediment. Ali and others (1998) reported that two species of chironomid (midge fly) larvae were highly susceptible to fipronil. Fifty percent of the test organisms died with 48 hours (LC 50) upon exposure to 0.42 µg/L.

The degradation products of fipronil also are toxic. Fipronil sulfone is 3.3 times more toxic to native bluegill sunfish than the parent compound. Fipronil sulfone is 6.6 times more toxic, and fipronil sulfide is 1.9 times more toxic to freshwater invertebrates than the parent compound (U.S. Environmental Protection Agency, 1996a).

McClain (2001) has shown that juvenile crawfish may be susceptible to the initial ricefield release water when fipronil-treated seed is applied following typical planting practices, and that water temperature and turbidity also may play a role in increased toxicity of fipronil to crawfish.

Table 1. Sampling sites for fipronil and degradation products, Mermentau River Basin, Louisiana

Site number (fig. 2)	Site name
1	Bayou Nezpique near Basile
2	Bayou Nezpique at Guidry Road near Basile
3	Bayou des Cannes near Eunice
4	Bayou Mallet near Eunice
5	Bayou Nezpique near Panchoville
6	Bayou des Cannes near Iota
7	Bayou Plaquemine Brule near Church Point
8	East Bayou Lacassine near Welsh
9	West Bayou Grand Marais near Roanoke
10	Mermentau River at Mermentau
11	Bayou Plaquemine Brule at Estherwood
12	Bayou Wikoff near Rayne
13	Bayou Chene near Thornwell
14	Bayou Lacassine near Lake Arthur
15	Mermentau River at Lake Arthur
16	Bayou Queue de Tortue at Riceville
17	Bayou Queue de Tortue near Lelieux

Fipronil in Surface Water

Concentrations of fipronil and three selected degradation products are listed in table 3. The maximum concentrations of the parent compound occurred in late March through mid-April. The maximum concentrations detected in this study were from the headwaters of small bayous surrounded by rice agriculture, such as 5.29 µg/L on March 23, at site 8 (East Bayou Lacassine near Welsh) and 5.19 µg/L on April 19, at site 9 (West Bayou Grand Marais near Roanoke). The maximum fipronil concentration detected at the main stem of the Mermentau River was 3.0 µg/L at site 10 (Mermentau River at Mermentau). The maximum concentration of fipronil occurs at or slightly before the maximum concentrations of other ricefield pesticides such as molinate (Goree and others, 2000, 2001).

Water samples also were analyzed for three fipronil degradation products (table 3). The degradation product detected in the highest concentrations was desulfinylfipronil. Desulfinylfipronil is the result of aerobic photodegradation, so the fact that this degradation product was detected in the highest concentrations in the water column (1.13 µg/L at site 7; 0.651 µg/L at site 2), as compared to the other two degradation products, conforms to

Table 2. Partitioning coefficients and fate of fipronil and three degradation products

Name	Company code no.[1]	Coefficient[2] K_{oc} (ave.)	Coefficient[2] log K_{ow}	Fate[1]
Fipronil	MB 46030	803	4.01	
Desulfinylfipronil	MB 46513	1,290	Not available	Photolysis in water or soil
Fipronil sulfide	MB 45950	2,719	Not available	Reduction in soil (anaerobic)
Fipronil sulfone	MB 46136	4,209	Not available	Oxidation in soil (aerobic)

[1]Rhone-Poulenc (1996); [2]Connelly (2001)

Table 3. Concentrations of fipronil and three degradation products in water, Mermentau River Basin, Louisiana, 2000

[Concentrations are in micrograms per liter; <, less than the method detection level; E, estimated; --, not analyzed]

Sample (month-day)	Fipronil	Desulfinyl-fipronil	Fipronil sulfide	Fipronil sulfone
Bayou Nezpique near Basile (site1)				
2-22	<0.0044	--	--	0.0062
3-21	1.29	0.151	0.054	.065
4-18	.593	.158	.109	.052
5-30	.028	.063	.148	.03
6-27	.009	.036	.087	.022
Bayou Nezpique at Guidry Road near Basile (site 2)				
2-22	<.0044	--	--	.012
3-21	3.94	.651	.067	.015
4-18	1.7	.242	.109	.089
5-30	.297	.082	.127	.049
6-27	.012	.059	.12	.037
Bayou des Cannes near Eunice (site 3)				
2-22	.005	--	--	.014
3- 9	.0044	--	--	.012
4- 5	.799	.166	.045	.05
4-18	2.69	.593	.124	.163
5-10	.088	.102	.076	.039
6-21	.014	.046	.058	.03
6-27	.016	.061	.081	.04
Bayou Mallet near Eunice (site 4)				
2-22	<.0044	--	--	.005
4-18	.829	.13	.043	.044
5-30	.298	.282	.043	.027
6-27	<.0044	.018	.022	.008
Bayou Nezpique near Panchoville (site 5)				
2-23	<.0044	--	--	.012
4-19	3.24	.36	.149	.143
5-31	.083	.079	.172	.05
6-28	.03	.071	.186	.05
Bayou des Cannes near Iota (site 6)				
2-23	.005	--	--	.013
3-22	2.69	.416	.071	.116
4-19	2.26	.413	.137	.126
5-31	.104	.171	.214	.07
6-28	.013	.071	.119	.038
Bayou Plaquemine Brule near Church Point (site 7)				
2-22	<.0044	--	--	<.005
3-21	1.59	1.13	.034	.126
4-18	.128	.036	.018	.011
5-30	<.0044E	.005E	.008	.004
6-27	<.0044E	.004E	.007	.004
East Bayou Lacassine near Welsh (site 8)				
2-24	<.0044	--	--	.019
3-23	5.29E	.2	.046	.132
4-19	4.54	.4	.171	.205
5-31	.09	.082	.163	.079
6-28	.024	.041	.1	.046
West Bayou Grand Marais near Roanoke (site 9)				
2-23	.007	--	--	.017
3-22	1.28	.12	.043	.066
4-19	5.19E	.425	.128	.202
5-31	.047	.066	.145	.057
6-28	.017	.033	.097	.037
Mermentau River at Mermentau (site 10)				
2-23	0.006	--	--	0.015
3- 8	<.0044	--	--	.013
3-23	.193	0.037	0.03	.018
4- 4	3.00	.51	.103	.163
4-19	1.02	.207	.071	.064
5- 9	.188	.068	.059	.038
5-31	.188	.101	.083	.044
6-20	.109	.109	.122	.052
6-28	.06	.086	.115	.052
Bayou Plaquemine Brule at Estherwood (site 11)				
2-23	<.0044	--	--	.013
3-22	3.04	.566	.085	.195
4-20	2.47	.541	.105	.144
6- 1	.243	.157	.129	.062
6-28	.019	.049	.066	.034
Bayou Wikoff near Rayne (site 12)				
2-23	<.0044	--	--	.004
3-21	.106	.091	.026	.018
4-19	1.07	.482	.044	.09
6- 1	.008	.038	.027	.015
6-27	<.0044E	.009	.012	.006
Bayou Chene near Thornwell (site 13)				
2-24	<.0044	--	--	.012
3-23	1.44	.09	.045	.066
4-20	2.6	.284	.122	.124
5-31	.086	.092	.107	.043
6-28	.047	.115	.169	.058
Bayou Lacassine near Lake Arthur (site 14)				
2-24	<.0044	--	--	.01
3-23	<.0044	.024	.011	.008
4- 4	1.94	.226	.077	.081
4-20	1.04	.219	.08	.068
5- 9	.176	.059	.048	.029
6- 1	.092	.073	.066	.029
6-20	.056	.094	.075	.033
6-28	.052	.121	.085	.042
Mermentau River at Lake Arthur (site 15)				
2-24	<.0044	--	--	.016
3-23	.09	.034	.028	.016
4-20	.915	.195	.064	.06
6- 1	.161	.112	.08	.046
6-29	.077	.1	.113	.053
Bayou Queue de Tortue at Riceville (site 16)				
2-24	<.0044	--	--	.01
3-23	1.64	.305	.037	.064
4-20	3.23	.534	.167	.194
6- 1	.282	.158	.14	.063
6-29	.016	.052	.084	.039
Bayou Queue de Tortue near Lelieux (site 17)				
2-25	<.0044	--	--	.009
3-24	4.07	.47	.069	.16
6- 1	.807	.236	.184	.11
6-28	.007	.03	.062	.024

the expected chemical behavior of the compounds in the environment. Interestingly, the highest desulfinylfipronil concentrations were detected at the same time as the highest fipronil concentrations at 14 of the 17 sites, and in the same months at 16 of the 17 sites (fig. 3). This indicates that the photolytic decomposition pathway is rapid.

The highest detected concentration of fipronil sulfide was 0.214 µg/L at site 6. The maximum concentration of fipronil sulfide tended to lag behind the maximum concentrations of the parent compound and the other two selected degradation products (fig. 3). This is consistent with the known degradation process, as fipronil sulfide is a reduction product most frequently detected in soils (U.S. Environmental Protection Agency, 1996a; Connelly, 2001).

The aerobic degradation product fipronil sufone had a similar detection pattern to the desulfinyl degradation product and the parent compound, with maximum concentrations occurring in March and April. The highest concentrations were 0.202 µg/L at site 9 and 0.205 µg/L at site 8. These are both wadable streams less than 20 feet in width. Aerobic degradation is more likely at these sites than in wider, deeper Mermentau Basin streams in which low dissolved oxygen concentrations are more common. Fipronil sulfone was the only degradation product analyzed in the February samples. Fipronil sulfone was detected at very low concentrations, about 0.01 µg/L, at 16 of the 17 sites in February, whereas the parent compound was detected only four times: 0.005 µg/L at site 3, 0.005 µg/L at site 6, 0.007 µg/L at site 9, and 0.006 µg/L at site 10. These concentrations were just above the minimum detection level of 0.0044 µg/L. The fipronil sulfone detected in February could be degradation products of fipronil application from the previous year, when fipronil was used on rice for the first time commercially.

Degradation product concentrations began to equal or exceed the parent compound in the May samples. In the June samples, only fipronil sulfone was detected in concentrations less than the parent compound at the downstream sites 10, 14, and 15.

Fipronil in Suspended Sediment

Fipronil and three degradation products in suspended sediment were determined from monthly samples at site 8, East Bayou Lacassine near Welsh, March-June and at site 14, Bayou Lacassine near Lake Arthur, March-May (fig. 4). Suspended-sediment concentrations of fipronil and degradation products range from 1 to 10 percent of the concentrations in water. Degradation product concentrations at site 8 exceeded the parent concentration by May, exhibiting the same general relation of parent compound to degradation products seen in the water column.

Fipronil in Bed Sediment

Fipronil in bed sediment (fig. 5) shows an interesting pattern. The parent compound was not detected in the 17 samples collected from August 28 to September 6, 2000. However, two degradation products, fipronil sulfide and desulfinylfipronil were detected at all 17 sites. The reduction product, fipronil sulfide, had the highest range of concentrations from 0.636 to 24.8 µg/kg (micrograms per kilogram). The photolysis product desulfinylfipronil concentrations ranged from 0.55 to 7.01 µg/kg. Fipronil sulfone was detected at 16 of the 17 sites and ranged from not detected to 10.5 µg/kg. Fipronil sulfone is an aerobic soil-oxidation product (U.S. Environmental Protection Agency, 1996a) and would be expected to occur in bed sediment.

The concentrations of the three degradation products in bed sediment also show a general increase downstream on all the streams, with the exception of Bayou Nezpique. The highest concentrations of all three degradation products were detected in bed sediment from the main stem of the Mermentau River at the two most downstream sites in the basin. In contrast, fipronil parent and degradation product concentrations in water at these sites generally were among the lowest during the February-June 2000 sampling.

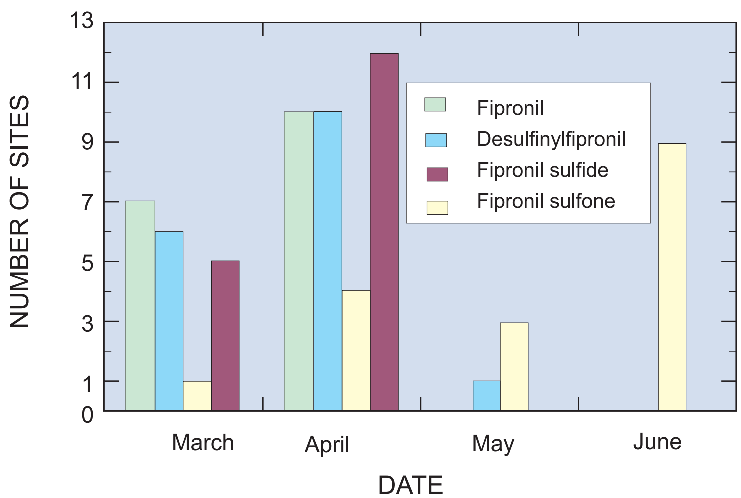

Figure 3. Occurrence of maximum concentrations of fipronil and three degradation products at 17 sites, March-June 2000.

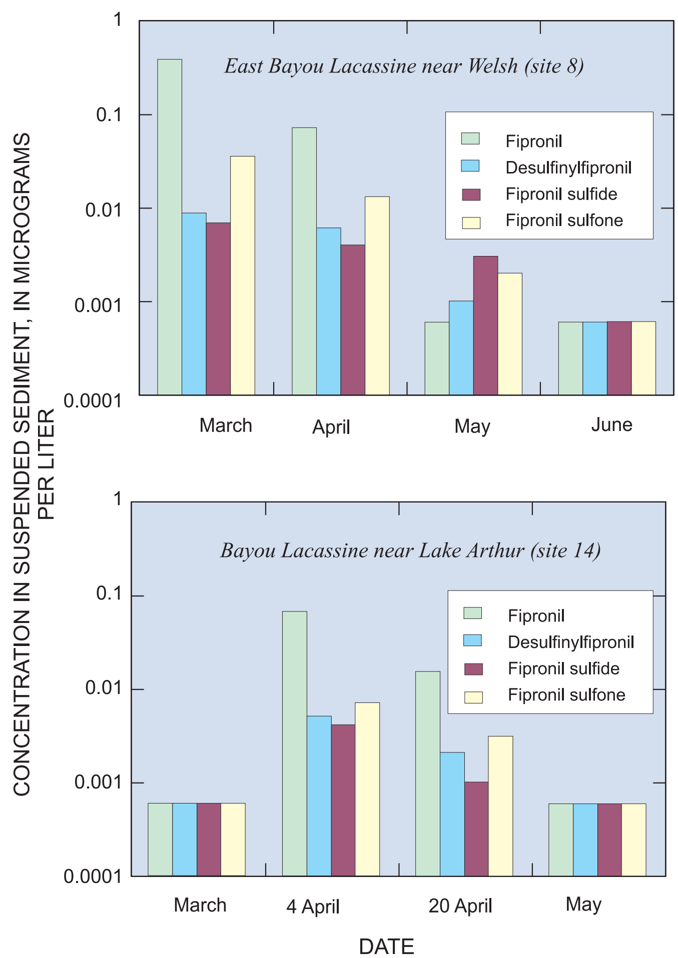

Figure 4. Fipronil and degradation products in suspended sediment, March-June 2000.

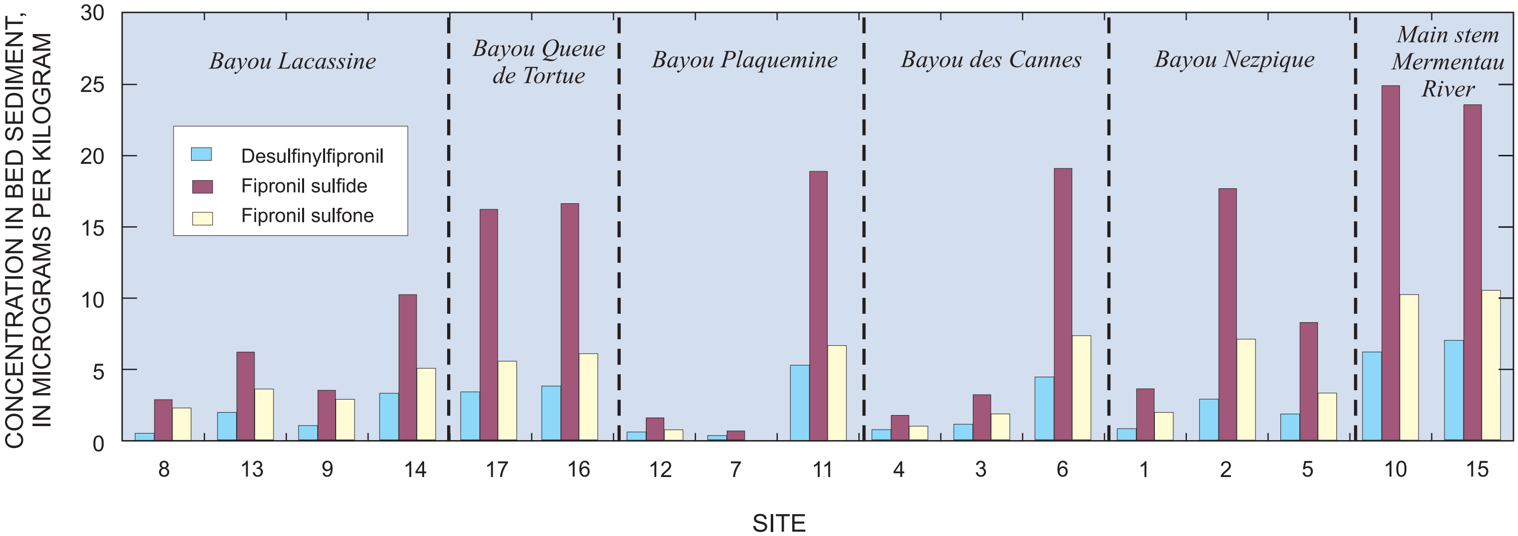

Figure 5. Fipronil degradation products in bed sediments, Mermentau River Basin, Louisiana, August 28-September 6, 2000.

ACKNOWLEDGMENTS

The authors wish to thank Bayer CropScience for providing additional reference standards of fipronil degradation products and James Madsen and Michael Schroeder of the USGS National Water Quality Laboratory for their work in the development of the custom method for the analysis of fipronil and degradation products.

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